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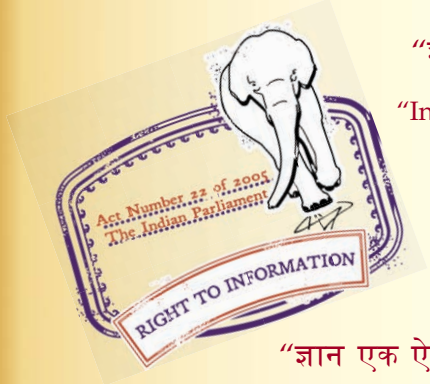
IS 6177 (2005): Phosphamidon Soluble Liquid (SL) [FAD 1: Pesticides and Pesticides Residue Analysis]



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"Knowledge is such a treasure which cannot be stolen"

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फॉसफामिडॉन विलेय द्रव — विशिष्टि

(दूसरा पुनरीक्षण)

Indian Standard

PHOSPHAMIDON SOLUBLE LIQUID (SL) — SPECIFICATION

(Second Revision)

ICS 65.100.10

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

October 2005

Price Group 2

FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Pesticides and Pesticides Residue Analysis Sectional Committee had been approved by the Food and Agriculture Division Council.

Phosphamidon soluble liquids are used in the control of insect pests of agricultural crops.

Soluble liquid formulation based on phosphamidon, technical, are generally manufactured to contain 40 percent (*m/m*) of phosphamidon.

This standard was first published in 1971. This standard has been revised to incorporate the latest packing and marking requirements.

In preparation of this standard, due consideration has been given to the provisions of the *Insecticides Act*, 1968 and the Rules framed thereunder. However, this standard is subject to the restrictions imposed under the *Insecticides Act* and Rules, wherever applicable.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

PHOSPHAMIDON SOLUBLE LIQUID (SL) — SPECIFICATION

(Second Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and the test for phosphamidon soluble liquids.

2 REFERENCES

The following standards contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
1070 : 1992	Reagent grade water (*third revision*)
4958 : 2005	Phosphamidon, technical
6940 : 1982	Method of test for pesticides and their formulations (*first revision*)
8190 (Part 2) : 1988	Requirement for packing of pesticides: Part 2 Liquid pesticides (*second revision*)
10627 : 1983	Methods for sampling of pesticides and their formulations

3 REQUIREMENTS

3.1 Description

The material shall be in the form of stable homogeneous liquid free from suspended matter. It shall consist of phosphamidon, technical dissolved in suitable solvent. A suitable dye shall be added to the formulation in small quantity to impart violet colour for safety purposes.

3.1.1 Phosphamidon, technical used in the preparation of the formulation shall conform to IS 4958.

3.2 Phosphamidon (Active Ingredient) Content

When determined by the method prescribed in Annex A, the observed phosphamidon content, of any of the samples shall not differ from the declared nominal value by more than percent tolerance limits indicated below:

Nominal Value Percent	*Tolerance Limit* Percent	
Up to 9	+ 10 − 5	of the nominal value
Above 9 and below 50	± 5	
50 and above	+ 5 − 3	

3.2.1 The actual value of the phosphamidon content in the formulation shall be calculated to the second decimal place for rounding off the first decimal place before applying the tolerances given in **3.2**.

3.2.2 The average content of all samples taken shall not be lower than the nominal value.

3.3 Acidity

When determined by the method prescribed in **13.5** of IS 6940, acidity (as H_2SO_4), if any, shall not be more than 1.5 percent by mass.

3.4 Solubility

The material shall be miscible with water in all proportions.

4 PACKING

The material shall be packed according to the requirements given in IS 8190 (Part 2).

5 MARKING

5.1 The container shall be marked legibly and indelibly with the following information:

a) Name of the material;

b) Name and address of the manufacturer;

c) Batch number;

d) Date of manufacture;

e) Date of expiry;

f) Net mass of contents;

g) Nominal phosphamidon content, percent (*m/m*);

h) The cautionary notice as worded in the *Insecticides Act*, 1968 and Rules framed thereunder; and

j) Any other information required under the *Standards of Weights and Measures (Packaged Commodities) Rules*, 1977.

5.2 BIS Certification Marking

The product may also be marked with the Standard Mark.

5.2.1 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

6 SAMPLING

When freshly manufactured material in bulk quantity and/or the retail pack of the formulated product is/are offered for inspection, representative sample of the material shall be drawn and tested as prescribed in IS 10627 and if tested within 90 days of its date of manufacture, the criteria for conformity shall be the content in percent (*m/m*) shall not be less than the declared nominal value. The upper limit for conformity shall be the same as those given in **3.2**.

When the material is offered for inspection after 90 days of its manufacture, sampling shall be done as prescribed in IS 10627. However, the criteria for conformity of the material, when tested, shall be the limit of tolerance, as applicable over the declared nominal value and given in **3.2**.

7 TESTS

7.1 Tests shall be carried out as prescribed in the appropriate methods given in **3.2** and **3.3**.

7.2 Quality of Reagents

Unless specified otherwise, pure chemicals and reagent grade water (*see* IS 1070) shall be employed in test.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

(*Clause* 3.2)

DETERMINATION OF PHOSPHAMIDON ACTIVE INGREDIENT CONTENT

A-0 GENERAL

Either of the two methods, namely, high performance liquid chromatographic (HPLC) and iodometric methods may be used for determining active ingredient content. However, in case of dispute HPLC method shall be the referee method.

A-1 IODOMETRIC METHOD

A-1.1 Outline of the Method

Phosphamidon along with by-products reacts with iodine in strongly alkaline solution consuming four equivalents of iodine. The by-products, but not phosphamidon, consume iodine in sodium carbonate solution. The differential titration thus gives the content of phosphamidon.

A-1.2 REAGENTS

A-1.2.1 *Standard Sodium Hydroxide Solution* — 2 N.

A-1.2.2 *Standard Sodium Carbonate Solution* — 2 N.

A-1.2.3 *Standard Hydrochloric Acid Solution* — 5 N.

A-1.2.4 *Standard Iodine Solution* — 0.1 N.

A-1.2.5 *Standard Sodium Thiosulphate Solution* — 0.1 N.

A-1.2.6 *Starch Solution* — 1 percent (*m/v*).

A-1.3 PROCEDURE

A-1.3.1 Weigh accurately about 8.6 g of sample into a distillation flask. Distill off the volatile organic solvent in a rotary evaporator at 60°C temperature and at about 15 mm of mercury pressure. Add 20 ml of water, mix thoroughly and distill off completely at 60°C temperature and at about 15 mm of mercury pressure.

A-1.3.2 Transfer the residue in the distillation flask to a 500-ml volumetric flask. Rinse the distillation flask with water several times, and add the washings to the solution in the volumetric flask and make up to volume with water (stock solution).

A-1.3.3 Pipette 10.0 ml of the solution into a 250-ml stoppered flask. Add, while stirring, 20.0 ml of the standard iodine solution. Then add 20 ml of the standard sodium hydroxide solution. Shake well and set aside in the dark at 25 ± 2°C for 30 min. Acidify the mixture with 20 ml of the standard hydrochloric acid solution and titrate the excess iodine with the

standard sodium thiosulphate solution using starch solution as the indicator. The end point is given by the discharge of blue colour. Note the value of titre (A ml). Carry out a blank with the same quantities of the reagents. Note the value of titre (V ml).

A-1.3.4 Pipette 10.0 ml of the stock solution into another 250-ml stoppered flask. Add, while stirring, 5.0 ml of the standard iodine solution. Add 5 ml of the standard sodium carbonate solution. Shake well and set aside in the dark at 25 ± 2°C for 15 min. Acidify with 5 ml of the standard hydrochloric acid solution and titrate as above with the standard sodium thiosulphate solution. Note the value of titre (B ml). Carry out a blank with the same quantities of the reagents. Note the value of titre (U ml).

A-1.4 CALCULATION

$$\text{Phosphamidon (active ingredient), percent by mass} = \frac{[(V-A)-(U-B)]}{74.93 \times f \times N} \over M$$

where

N = normality of sodium thiosulphate solution;

M = mass of the sample taken for the test, in g; and

f = correction factor.

A-2 HIGH PERFORMANCE LIQUID CHROMATOGRAPHIC METHOD

A-2.1 Outline of the Method

Phosphamidon is determined by reverse phase high-pressure liquid chromatography. Test sample of phosphamidon is compared with phosphamidon reference standard and percentage of phosphamidon is estimated by external standard method.

A-2.2 Apparatus

A-2.2.1 *HPLC System* — Liquid chromatograph equipped with injector (10-microlitre loop), high sensitivity UV detector and suitable electronic integrator.

A-2.2.2 *Chromatographic Column* — RP-18; particle size : 5 µm; length : 250 mm; inner diameter : 4.6 mm or equivalent.

A-2.3 Reagents

A-2.3.1 *Acetonitrile* — HPLC grade.

A-2.3.2 *Water* — HPLC grade.

A-2.3.3 *Phosphamidon Standard* — of known purity.

A-2.3.4 *Mobile Phase* — Acetonitrile and water, 42:58 *v/v*.

A-2.4 Operating Conditions

A-2.4.1 *Eluent Flow Rate* — 1 ml/min.

A-2.4.2 *Temperature* — ambient.

A-2.4.3 *Injection Volume* — 10 microlitre, fixed loop.

A-2.4.4 *Detector Wavelength* — 220 nm.

A-2.5 Procedure

A-2.5.1 *Preparation of Reference Standard Solution*

Weigh (to the nearest of 0.1mg) into a volumetric flask (250 ml) sufficient reference sample to contain 100 mg of phosphamidon (W_s). Dissolve and dilute it up to the mark with mobile phase.

A-2.5.2 *Preparation of Test Sample Solution*

Weigh (to the nearest of 0.1mg) into a volumetric flask (250 ml) sufficient sample to contain 100 mg of phosphamidon (W_t). Dissolve and dilute it up to the mark with mobile phase.

A-2.5.3 *System Equilibration*

Inject 10 micro litre of the reference standard solution till areas of phosphamidon peak from two consecutive injections agree to 1 percent maximum.

A-2.5.4 *Determination*

Make duplicate 10 micro litre injections of the sample solution, followed by injection of reference standard sample. Average the areas of the injections following and preceding the sample injection (A_s). Record the relevant peak areas of the sample (A_t).

A-2.5.4.1 *Guide values*

Retention time — 4.8 min (phosphamidon peak — *see* Note).

Run time — 21 min.

NOTE — Phosphamidon peak may split (isomer separation) depending upon the condition of analytical column. In that case add both the peak areas for calculation purpose. Retention time is an indicative parameter that may change slightly time to time.

A-2.6 Calculation

$$\text{Phosphamidon content, percent by mass} = \frac{W_s \times A_t \times P}{W_t \times A_s}$$

where

W_s = weight of reference standard sample,

W_t = weight of test sample,

A_t = average area of phosphamidon peak in the test solution,

A_s = average area of phosphamidon peak in the reference standard solution, and

P = percent purity of reference standard sample.

NOTE — Wash the column by methanol, followed by chloroform and again by methanol (50 ml for each solvent) after analysis.

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act,* 1986 to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards : Monthly Additions'.

This Indian Standard has been developed from Doc : No. FAD 1 (1395).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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Printed at New India Printing Press, Khurja, India